



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/659,577 Confirmation No. 4666
Applicant : Lawrence T. Drzal and Hiroyuki Fukushima
Title : EXPANDED GRAPHITE AND PRODUCTS PRODUCED
THEREFROM
Filed : September 10, 2003
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Customer No. : 21036

Mail Stop RCE
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.132

Dear Sir:

Lawrence T. Drzal states as follows:

(1.) That I am an inventor of the invention in the above entitled application.

(2.) That I am the Director of the Composite Materials & Structures Center and a Distinguished Professor of Chemical Engineering and Materials Science and Mechanics at Michigan State University, East Lansing, Michigan 48824.

(3.) That my research and field of expertise is directed to nanomaterials (graphene nano-platelets) as multifunctional modifiers for polymers.

(4.) That unlike the teachings of U.S. Patent No. 6,024,900 to Saito *et al.*, the precursor graphite in the above entitled application is expanded within a microwave or radiofrequency wave applicator.

(5.) That the following explains the difference between conventional heating and microwave or radio frequency heating. This explanation also directly explains why the structure implied by microwave heating significantly improves and is different than the structure achieved through conventional heating.

(6.) Conventional heating of intercalated graphite relies on convective and conductive heat transfer. Conductive heat transfer occurs from the outside to the inside of intercalated graphite to rapidly raise the temperature of the intercalated acid to above its boiling point in order to vaporize it. The intercalated acid vapor is partially confined between the graphene sheets. The acid vapor can leak out between the sheets along the edges of the platelets while the temperature of the acid vapor is increasing. The vapor pressure increases and eventually forces the platelets apart causing a volumetric expansion.

(7.) Microwave heating relies on coupling of electromagnetic radiation to the intercalated graphite and the intercalated acid. The degree of coupling depends on the properties of the graphite and the acid. The dielectric

constant of graphite is around 12-15 while that of sulfuric acid is around 100. This means when sulfuric acid intercalated graphite is exposed to microwave radiation; the acid inside the graphite absorbs more microwave energy and heats up faster than the graphite, as long as the microwave has enough energy to penetrate the graphite layers. This method of heating is faster since it does not rely on the slower conventional conduction and convection heating mechanisms. Microwave heating as a result leads to a greater degree of expansion over conventional heating since the acid intercalated inside the graphite can quickly increase in temperature above the boiling point, and then vaporize at a much faster rate than conventional heating. As a result the leakage of acid from the edges of the intercalated graphite does not increase to a faster rate leading to a much higher degree of expansion.

(8.) For example, a 10g sample of commercially available acid intercalated graphite, Asbury Expandable Graphite Grade 3772, was expanded in a conventional oven at 1000°C. [Sample A] Also the same amount of the same sample was expanded by 915 MHz microwave at 10 kW. [Sample B] The surface area of sample B after microwave processing was 55 m²/g while that of sample A was 33 m²/g. Since the surface area after the expansion is an indicator of the degree of exfoliation, this result clearly shows that microwave process can exfoliate acid intercalated graphite more efficiently than a conventional heating process. Moreover, the improvement in expansion is not limited to this particular example. As a result of the mechanism described above, microwaving acid intercalated graphite will result in better expansion and thus a different structure than that of conventional heating.

(9.) It is further understood that one skilled in the art would not consider microwaving as an obvious alternative to conventional heating. This is especially true in the field of graphite expansion. The graphite is a conductive material. It is generally known that conductive materials are typically

unsuitable for microwave heating since the electromagnetic stimulation can cause undesired sparking and potential explosions. However, when small discrete conductors such as the intercalated graphite particles are in a microwave environment, they heat-up rapidly through a resistive heating mechanism. Thus, general knowledge in the art would teach and suggest away from trying microwaving to heat and boil a precursor chemical intercalated in a graphite material.

(10.) The undersigned declares further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.


Lawrence T. Drzal

Date: April 13, 2009